

MASSACHUSETTS INSTITUTE OF TECHNOLOGY
RESEARCH LABORATORY OF ELECTRONICS
Cambridge, Massachusetts 02139

Reprinted from

Quarterly Progress Report No. 104, January 15, 1972

**CASE FILE
COPY**

The Research Laboratory of Electronics is an interdepartmental laboratory in which faculty members and graduate students from numerous academic departments conduct research. The research reported in this document was made possible by support extended the Massachusetts Institute of Technology, Research Laboratory of Electronics, by the following agencies.

Joint Services Electronics Programs (U. S. Army, U. S. Navy, U. S. Air Force)

Contract DAAB07-71-C-0300

U. S. Army - Research Office - Durham

Contract DAHC04-71-C-0039

Contract DAHC04-69-C-0042

U. S. Navy - Office of Naval Research

Contract N00014-67-A-0204-0019

U. S. Air Force - Electronics Systems Division

Contract F19628-69-C-0044

Contract F19628-70-C-0064

U. S. Air Force - Office of Scientific Research

Contract F44620-71-C-0051

U. S. Coast Guard Contract DOT-CG-13446-A

National Aeronautics and Space Administration

Grant NGL 22-009-013

Grant NGL 22-009-337

Grant NGR 22-009-526

Grant NGL 22-009-016

Grant NGR 22-009-091

Contract NAS1-10693

Grant NGL 22-009-304

Grant NGR 22-009-421

Contract NAS8-27733

National Institutes of Health

Grant 5 PO1 GM14940-05

Grant 5 TO1 GM01555-05

Grant 5 RO1 NS04332-09

Grant 3 PO1 GM15006-03S2

Grant 5 TO1 HD-00111-07

National Institute of Mental Health Grant 5 PO1 MH13390-5

National Science Foundation

Grant GK-28282X

Grant GP-14589

Grant GP-21348

Grant GP-28679

Grant GP-20769

Grant GP-24254

National Bureau of Standards Grant 1-35857

U. S. Atomic Energy Commission

Contract AT(11-1)-3070

Bell Telephone Laboratories, Incorporated Grant

World Health Organization Grant R/00348 and Associated Press Grant

Boston City Hospital Purchase Order 10656 and B-D Electrodyne Division Grant

M. I. T. Lincoln Laboratory Purchase Orders CC-544, CC-570, A-6270

M. I. T. Sloan Fund for Basic Research Grants 241 and 616

California Institute of Technology Contract 952568

Support of projects is acknowledged in footnotes to the appropriate sections.

THIS DOCUMENT HAS BEEN APPROVED FOR PUBLIC
RELEASE AND SALE; ITS DISTRIBUTION IS UNLIMITED.

V. SOLID-STATE MICROWAVE ELECTRONICS*

Academic Research Staff

Prof. P. L. Penfield, Jr.
Prof. D. H. Steinbrecher

Graduate Students

A. Chu	M. S. Navarro
G. P. Hom	Y. K. Yee

RESEARCH OBJECTIVES

Our research objectives include developing fundamental performance limits and designing optimum embedding networks for diodes in microwave applications.

During the coming year we plan to investigate the application of the Josephson junction to problems of low-noise millimeter wave detection. In this case the frequency range of interest is 100-600 GHz: We plan to take an approach similar to that of millimeter-wave mixer diode development that has been in progress for several years.

In other areas we are seeking to establish the fundamental limits on intermodulation distortion in PIN diodes. We shall investigate workable embedding for MSM bulk-effect devices that will yield low-noise amplification in the microwave region, and also investigate circuit techniques for the design of extremely wideband low-noise IF amplifiers in the range 1-1000 MHz with a noise figure under 2 dB.

P. L. Penfield, Jr., D. H. Steinbrecher

A. PEAK-LOCKING APPROACH FOR STABILIZATION OF HIGH-RESOLUTION MASS SPECTROMETERS†

1. Introduction

Much effort has been devoted toward improving resolution and stability in the CEC 21-110, Mattauch Herzog double-focusing mass spectrometer. At present, measurements indicate that magnetic-field instability is the major source of performance degradation.

This report first reviews the stringent requirements imposed by some experiments that the present instrument is incapable of meeting, and then I discuss a control system based on locking a mass peak by introducing compensatory corrections in the magnetic field.

*This work was supported by the National Aeronautics and Space Administration (Grant NGL 22-009-304) and by the Joint Services Electronics Programs (U.S. Army, U.S. Navy, and U.S. Air Force) under Contract DAAB07-C-0300.

†Supported by National Institutes of Health Grant RR00317 from the Biotechnology Resources Branch, Division of Research Resources.

(V. SOLID-STATE MICROWAVE ELECTRONICS)

2. Experiments Requiring Operation of the Present Mass Spectrometer at and beyond the Limits of Performance

a. Analysis of Target Compound A at Concentrations of Less Than 1 ppm in a Volatile Mixture B for Sample Sizes in the Range 0.3-1 mg

Detection of insecticides, herbicides and other toxic compounds in the environment requires these, and even greater, detection limits, if possible. If 1 ppm of A in B is detectable during a 3-min exposure time, then 0.1 ppm is detectable in a 30-min exposure.

b. Analysis of Volatile Compounds Trapped in a Nonvolatile Matrix

The detection of oil residues in oil shale, volatiles in lunar samples, and so forth requires heating a powdered sample at high temperatures for periods over 10 min. Any instrument instabilities resulting in a mass measurement error greater than 5 ppm will prevent accurate mass assignment of elemental compositions.

c. Analysis of Compounds with High Molecular Weight (1000 amu)

During the analysis of compounds with high molecular weight any electric or magnetic field drift results in loss of mass accuracy and causes a consequent loss in our ability to assign elemental compositions. If the compound in question is $C_{62}H_{120}N_4O_5$, and the measured mass is 1000.9258, then a unique and correct elemental composition can be assigned, provided that the relative error of the mass measurement is 1 ppm. If the mass measurement error resulting from instrument instability is degraded to 5 ppm, then 3 elemental compositions are possible; for 10 ppm, the number of possible compositions is 8. Furthermore, an increase in the number of heteroatoms N_4O_5 to a larger number $N_8O_{15}Si_2$ causes a tremendous increase in the number of possibilities.

d. Direct Analysis of Low-Volatility Compounds

Direct analysis of low-volatility compounds is limited by the instrument instabilities during long vaporization periods. Therefore conversion of the compound into a more volatile derivative (with loss in structure information) is necessary.¹ The vaporization times for a direct analysis are in the range 1-24 h.

3. Previous Work and Present State of the Mass Spectrometer

Upgrading of the manufacturer's specifications required theoretical studies,² a controlled environment, and improvements in instrumentation. The instrument was placed in a semiclean electrically shielded room³ with temperature and humidity controls. It was mounted on a 1-Hz air piston antishock and vibration-controlled assembly.

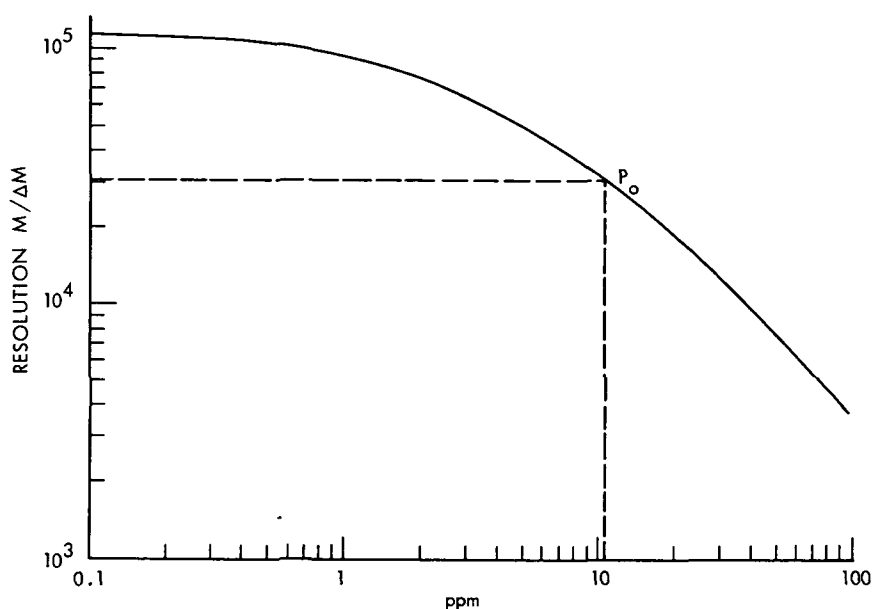


Fig. V-1. Degradation of resolution with random power supply error.
Exit slit = $2.5 \mu\text{m}$; theoretical resolution = 1.25×10^5
(absence of power supply noise); operating point P_o determined by measurement.

Power-line isolation in excess of 150 dB from 10 Hz to 1 GHz had been reported by Lovins and Steinbrecher.³ For the electric sector power supply we substituted two precision DC Standards (HP 740-B) with a stability of 1 ppm.

As expected, power-supply noise and stability measurements placed the operating point at P_o (stability, 10 ppm, resolving power, 30,000) as illustrated in Fig. V-1. This indicates that the performance can indeed be improved by stabilizing the magnetic field, since the electric field stability is 1 ppm with the mentioned substitution.

A stability figure for magnetic field which is compatible with the electric field stability is 1 ppm.

4. Peak-Locking Approach

Stabilization of each field (in the range of a few parts per million, over 1-10 h periods) by using feedback circuits with dc voltage references, strikes against the flicker-noise problem, which imposes degradation when time periods are extended. Consequently, an alternative approach will be taken.

Our purpose is to obtain a peak that is fixed in space within such limits that, for the instrument operating with high resolution, the peak stability is not a degrading factor of resolving power. A control system that introduces changes in the magnetic field to maintain the position of a given mass peak will be considered. The peak-locking approach will stabilize the entire system with direct control of mass accuracy.

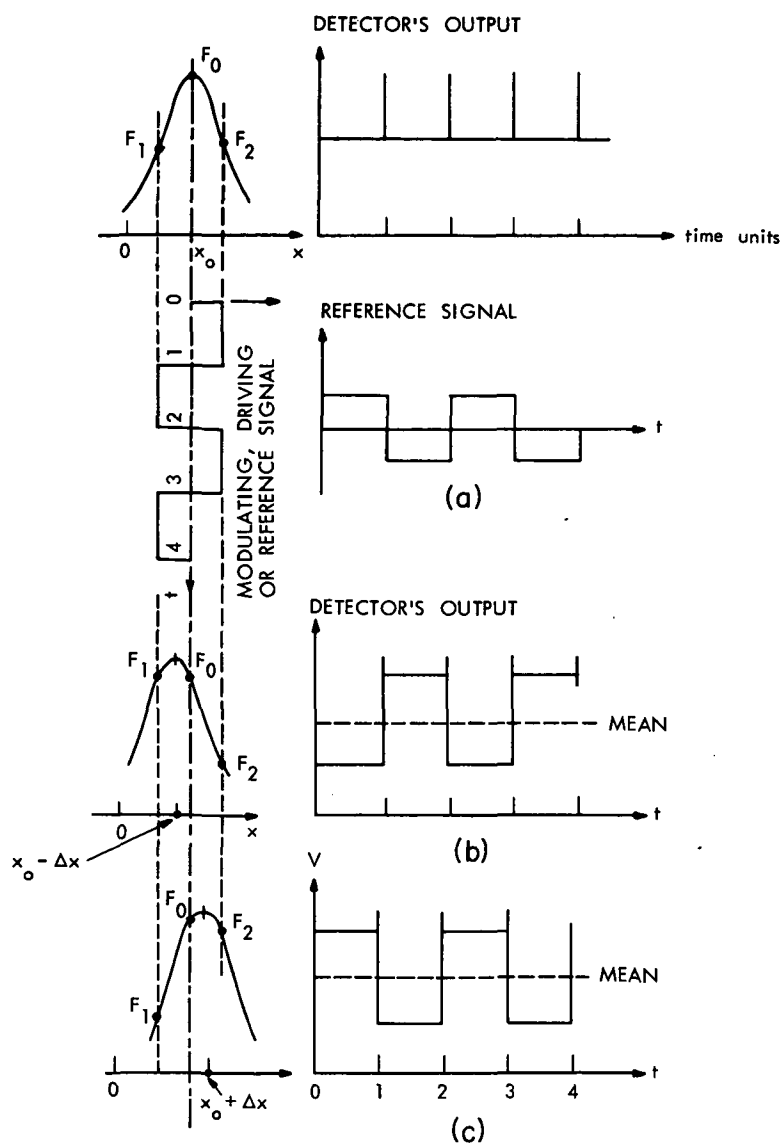


Fig. V-2. Principle of operation.

- (a) Lock on a mass peak.
- (b) Peak drifts to the $-x$ direction.
- (c) Peak drifts to the $+x$ direction.

a. Principle of Operation

Simultaneous photoplate detection and electrical single-peak detection are possible. A reference peak can be located with respect to the exit slit so that the electron multiplier is exposed to the maximum F_0 of the ion flux distribution; see Fig. V-2a. Based on the symmetry of the mass peak, if the ion beam is modulated with a symmetrical square wave, the detector will read the ion fluxes at F_1 and F_2 . The corresponding output voltages will be identical, provided the peak does not drift. Therefore the output

signal of the detector, in arbitrary units proportional to the ion flux, will be a dc value with superimposed spikes resulting from the excursion from F_1 to F_2 through F_0 , in time interval (t^-, t^+) . Figure V-2b shows the consequences of a drift of the flux profile in the $-x$ direction. The detector's output corresponding to the exposures to F_1 and F_2 is no longer equal and the resulting output is a square wave with mean and superimposed spikes. By the same procedure, the output signal for a drift in the $+x$ direction can be constructed as shown in Fig. V-2c. Inspection of the output waveforms in Fig. V-2 and the reference modulation signal leads us to the following conclusions.

- (i) In the absence of drift the output of the detector is a dc value.
- (ii) A drift in the $\begin{pmatrix} +x \\ -x \end{pmatrix}$ direction produces a $\begin{pmatrix} 0 \\ 180^\circ \end{pmatrix}$ phase shift ϕ with respect to the reference square-wave signal.
- (iii) The magnitude of the square wave, and hence the amplitude of the first harmonic, is a monotonic function of the drift.

A simulation carried out by using a set of deflection plates that are available in the existing detector and leaving the exit slit wide open confirmed the conclusions.

b. Block Diagram

A "lock-in amplifier" will be used to process the information contained in the detector's output and the reference signal. The output of the lock-in amplifier locked on the fundamental of the square wave is

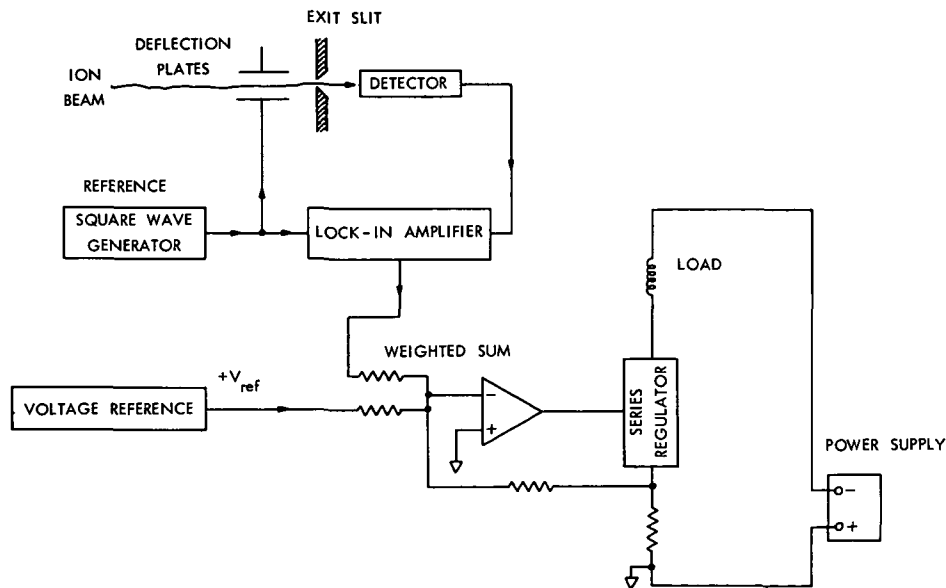


Fig. V-3. Diagram of peak-locking control.

(V. SOLID-STATE MICROWAVE ELECTRONICS)

$$E_{\text{out}} = \frac{2}{\pi} E \cos \phi \quad \begin{cases} +\frac{2}{\pi} E & \text{for a } +x \text{ drift} \\ -\frac{2}{\pi} E & \text{for a } -x \text{ drift} \end{cases}$$

This output will be weighted with the reference voltage of the control circuit (see Fig. V-3).

c. Performance

A voltage reference was built, and indirect stability measurements indicated a stability better than 1 ppm in a controlled environment. Power-supply isolation in excess of 70 dB was measured for frequencies up to 100 kHz. With this reference the magnetic field stability should be approximately 1 ppm; therefore, with the peak-locking control incorporated, the performance in mass accuracy should be at least 1 ppm over the depletion time of the reference peak, which can be chosen to be longer than the depletion time of the target compound. At present the magnet stability is somewhere between 5 ppm and 10 ppm for a 10-min period.

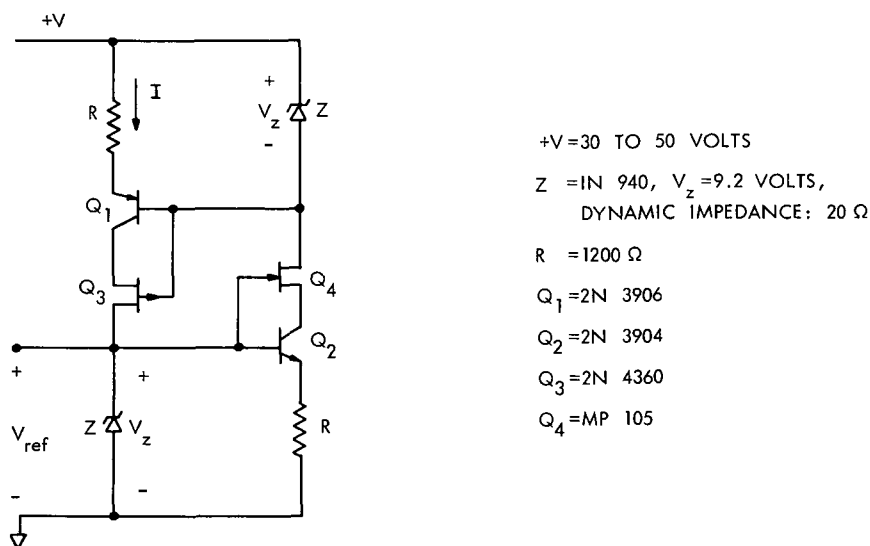


Fig. V-4. Reference voltage circuit.

5. Voltage Reference

A voltage reference circuit⁴ was built (see Fig. V-4). The components were imbedded in an aluminum heat sink. The Zener current stability was 15 ppm per hour. The voltage stability is given by

(V. SOLID-STATE MICROWAVE ELECTRONICS)

$$\frac{\Delta V_z}{V_z} = \frac{R_d \Delta I}{V_z} + \frac{\partial V}{\partial C} \Delta C \quad V \approx RI$$

$$\frac{\Delta V_z}{V_z} = \frac{R_d}{R} \frac{\Delta I}{I} + \frac{\partial V}{\partial C} \Delta C,$$

where

$$R_d = 20 \, \Omega \text{ (dynamic impedance)}$$

$$R = 1200 \, \Omega$$

$$\frac{\partial V}{\partial C} = 2 \times 10^6 / ^\circ\text{C}$$

$$\Delta C = 0.1^\circ\text{C}.$$

Substitution yields a voltage stability of 0.45 ppm per hour.

6. Further Work

We are planning a computer analysis of the control circuit to evaluate design and performance, and modification of the three-way slit to incorporate a set of deflection plates.

A. Chu

References

1. E. Vilkas and E. Lederer, "Application of the Hakomori Method to the Permethyla-tion of Peptides," Tetrahaedron Letters 3089 (1968); see also D. Junt, C. Hignite, and K. Biemann, "Structure Elucidation of Dinucleotides by Mass Spectrometry," Biochem. Biophys. Res. Communs., Vol. 33, No. 3, 1968.
2. J. I. Glaser, Quarterly Progress Report No. 87, Research Laboratory of Electron-ics, M.I.T., October 15, 1967, pp. 17-24; Quarterly Progress Report No. 90, July 15, 1968, pp. 24-35.
3. R. E. Lovins and D. H. Steinbrecher, Quarterly Progress Report No. 88, Research Laboratory of Electronics, M.I.T., January 15, 1968, pp. 36-37.
4. P. Williams, "A DC Reference Voltage with Very High Rejection of Supply Varia-tion," Proc. IEEE 56, 118-119 (1968).